

*good for
100 runs*

TEFLON* FEP-FLUOROCARBON RESIN, AQUEOUS DISPERSION

Determination of Equivalent ^{C-8 or} C-9 Dispersing Agent

I. Applicability

This method is applicable to determining the equivalent ^{C-8 or} C-9 dispersing agent in FEP dispersion.

II. Unusual Safety Considerations

III. Principle

Percent C-9 is determined by titrating FEP dispersion with a solution of cetyltrimethylammonium bromide.

IV. Sensitivity, Precision, and Accuracy

The precision (sigma) of this test based on five runs made on one sample is 0.0005 with an $\bar{X}$ of 0.1254% C-9.

V. Reagents

1. Cetyltrimethylammonium bromide (CTMAB), 1 g/liter (RC 190.127)
(HEXADECYLTRIMETHYL AMMONIUM BROMIDE)

VI. Procedure

1. Weigh 25 ± 0.01 g FEP dispersion into a 600-ml beaker.
2. Add 200 ml (grad cyl) demineralized water to the dispersion.
3. Stir the beaker using a magnetic stirrer until the dispersion and water are mixed (ca. 30 seconds).
4. Fill a buret with 1 g/liter CTMAB solution. Record the reading.
5. Using the buret, add CTMAB solution to the dispersion slowly, with constant magnetic stirring. As CTMAB is added, polymer will begin to coagulate. When it appears that most of the polymer has coagulated, stop adding CTMAB and then stop agitation a few seconds later.
6. Allow the bulk of the polymer to settle out and observe the supernatant liquid. If it is cloudy, resume agitation and add 0.2 to 0.5 ml of CTMAB.
7. Stop agitation and allow to settle.

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8. Repeat steps 6 and 7 until the supernatant liquid is perfectly clear. A clear supernatant liquid is the end point of the titration.
9. Record the buret reading.
10. Find specific gravity of the dispersion and percent solids, using Method T 202.804.
11. Calculate % C-9 and g C-9/g FEP.

VII. Calculations

1. Calculation of % C-9

ml CTMAB = (step 9 - step 4)

$$\% \text{ C-9} = \frac{\text{ml CTMAB}}{1000} \times \frac{463}{364} \times \frac{100}{\text{wt of dispersion}}$$

$$= 0.1272 \times \frac{\text{ml CTMAB}}{\text{wt of dispersion}}$$

where:

molecular wt C-9 = 463
molecular wt CTMAB = 364

2. Calculation of g C-9/g FEP

% FEP = % solids (step 10)

% C-9 from step 1, Calculations

$$\frac{\text{g C-9}}{\text{g FEP}} = \frac{\% \text{ C-9}}{\% \text{ FEP}}$$

For C-8 replace 463 with 431

$$\text{For \% C-8: } 0.1184 \times \frac{\text{ml CTA}}{\text{DISP. W}}$$

VIII. References

1. Couture, M. J., Washington Works Technical Laboratory Notebook No. 1026, pp 88-89
2. Plastics Department Laboratory Test Method T 202.804
3. Original Method, Washington Works Technical Laboratory, 3-11-71

Revised to reduce sample size to give better end point and to specify demineralized instead of deionized water.

Origin: Washington Works Technical Laboratory, 1st Circulation 12-11-75
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Approved by Laboratory Methods Coordination Committee, 2-16-76

WW-2939

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